ON THE VAPORIZATION AND THERMAL OXIDATION OF CHLORINATED HYDROCARBON/ALCOHOL SPRAYS

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We report species concentration data obtained during the vaporization and thermal decomposition of polydisperse multicomponent chlorinated hydrocarbon (CHC)/alcohol sprays. Mixtures of 1,1,1-trichloroethane ($C_2H_3Cl_3$) (TCA) and isopropanol (C_3H_7OH) are atomized in the postflame environment of a methane/air ($\Phi = 0.95$) flame. Species concentrations, determined by extractive Fourier transform infrared analysis, reveal perturbations in TCA destruction and by-product species formation. The effect of isopropanol addition on by-product species concentrations is similar to previous results involving the thermal oxidation of TCA/alkane mixtures. TCA destruction is incomplete at injection temperatures less than 1000 K. Under these conditions, TCA/isopropanol sprays produce lower residual TCA concentrations than pure TCA sprays. Review of physical property data, numerical modeling (to be published elsewhere), and comparisons with previous experimental results for TCA/heptane and TCA/hexadecane together suggest two primary findings. First, the conventional approach of parameterizing fuel volatility by the fuel saturation temperature at 101.3 kPa does not correlate with the trends evident in our data. For these fuel mixtures and conditions, the latent heat of vaporization (ΔH_v) provides a better correlation. Second, the data also suggest that, for incineration-resistant multicomponent mixtures, the molecular weights of mixture constituents may influence droplet combustion characteristics and should be considered when formulating waste-blending strategies.

Background

Fuel injection plays an important role in liquid hazardous waste incineration. Liquid waste incineration faces some of the same challenges as conventional combustion processes, including the impact of chemical kinetics on combustion efficiency and emissions, multicomponent fuels, fuel/air mixing, and surface quenching. Hazardous waste incinerators must meet these challenges while adhering to strict waste destruction removal efficiency and emissions standards. Fuel atomization and vaporization in particular are crucial in achieving sufficient fuel/air mixing and complete combustion. Waste composition can vary widely, enough so that characterization by standard waste classification codes is difficult [1]. As a result, mean properties of a mixed waste stream are difficult to evaluate for the purposes of selecting appropriate incinerator operating conditions. Adequate data are not available regarding the impact of process upsets on incinerator performance [1]. Results from Environmental Protection Agency (EPA) bench- and pilot-scale facilities have indicated little change in the destruction of the principal organic hazardous constituent during simulated upset conditions. However, other investigators [2–4] have reported significant changes in the products of incomplete combustion during bench-scale simulations of incinerator process upsets. The influence of variable waste composition and process upsets on incinerator performance is of particular concern to operators of waste cofiring facilities. Cofiring allows waste-generating industries to burn hazardous waste–derived fuels (HWDFs), mixtures of liquid hazardous wastes and conventional hydrocarbon fuels, at the same facility. This distributes the public burden of siting incinerators and reduces the costs of hazardous waste treatment. The number of facilities using HWDFs was over 1300 in 1984 and over 900 in 1991 [1]. Examples of such industries include chemical, paper, petroleum, concrete, and primary metals.

Most combustion processes for power generation and propulsion applications use multicomponent liquid hydrocarbon fuels. Studies of multicomponent droplet vaporization have therefore been both a practical necessity and a natural extension of single-component results. The temporally and spatially

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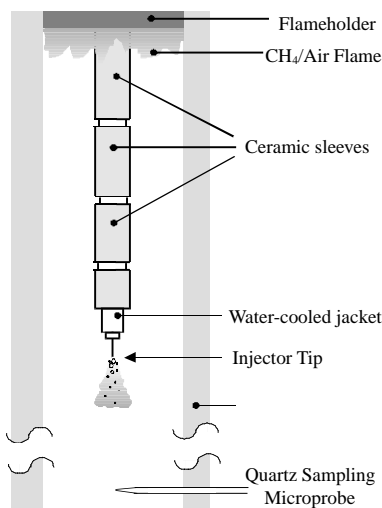| 16. SECURITY CLASSIFICATION OF:			17. LIMITATION OF ABSTRACT	18. NUMBER OF PAGES	19a. NAME OF RESPONSIBLE PERSON
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FIG. 1. Schematic of combustion driven flow reactor (drawing not to scale).

TABLE 1
Selected thermodynamic properties of 1,1,1-trichloroethane (TCA) ($C_2H_3Cl_3$), isopropanol (C_3H_7OH), heptane (C_7H_{16}), and hexadecane ($C_{16}H_{34}$) [22]

Property	TCA	C_3H-OH	C_7H_{16}	$C_{16}H_{34}$
Density (kg/m^3)	1349.2	785.1	684	773.5
Molecular weight (kg/kmol)	133.4	60.1	100.2	226.4
T_b^0 (K)	347	356	372	560
C_p (at 300 K) (J/mol K)	144	155–161	224.7	500
ΔH_v (at 298 K) (kJ/mol)	32.6	45.5	36.7	81

varying mixture composition within a droplet undergoing transient heating underlies much of multicomponent droplet vaporization behavior and forms the focus of many investigations. Megaridis [5] and Sirignano and coworkers [6–9] reported numerical results from computational models incorporating variable liquid properties, internal convective motion, surface blowing, droplet deceleration, and surface regression. Randolph et al. [10] sampled vaporizing bicomponent droplets and reported on the influence of liquid-phase mass diffusion for vaporizing droplets at $Re \approx 0$. For mixtures of fuels that have large differences in volatility, rapid heating can lead to nucleate boiling of the more volatile component in the droplet interior. The potential then exists for the spontaneous rupture of the droplet by the expanding vapor ("microexplosions"). Law [11] was among the first to investigate this potential, and several authors

have subsequently reported experimental evidence of microexplosions [12–14]. However, the numerical results of Megaridis and Sirignano [8,9] indicate no possibility of microexplosions for the mixtures and conditions they investigated.

Droplet processes are also important in the thermal treatment of liquid hazardous wastes. Law [15] reviewed how atomization and droplet vaporization phenomena influence hazardous waste incineration. The multicomponent nature of liquid hazardous waste inventories makes multicomponent droplet effects an inherent part of waste incineration. However, various investigators have sought to exploit multicomponent effects to improve the relatively poor combustion characteristics of incinerable wastes, particularly chlorinated hydrocarbons (CHC). These characteristics can lead not only to incomplete waste destruction but also to the formation of toxic combustion by-products in the event of a process upset. Results by Sorbo and coworkers [19] and Sorbo and Chang [20] indicate that droplets composed of 75/25 volume percent CHC/alkane mixtures result in increased burning rates as compared with pure CHC droplets. However, Thomson et al. [4] reported that similar CHC/alkane mixtures did not improve in the destruction of CHCs in a postflame environment. Our past investigations of 1,1,1-trichloroethane (TCA) thermal oxidation [2,3] indicated either enhanced or suppressed waste destruction, depending on the spray composition, spray density, or spray mean droplet size. In particular, results from Ref. [3] indicate that spray vaporization phenomena influence both TCA destruction and by-product species formation for mixtures of TCA/heptane and TCA/hexadecane in a lean postflame environment. The current study examines the complementary experimental conditions of TCA/oxygenate sprays thermally decomposing within a near-stoichiometric postflame environment.

Experimental Design

Solid cone liquid sprays of pure TCA and TCA/isopropanol (C_3H_7OH) mixtures (75/25 by volume) were separately introduced into the postflame environment of a vertically oriented combustion-driven (methane/air) flow reactor (Fig. 1). Extractive sampling using an uncooled quartz microprobe occurred 1.22 m downstream of injection. Gas samples, analyzed using long-path Fourier transform infrared spectroscopy [18], provide plots of species concentration versus injection temperature. The chosen oxygenate was isopropanol, because its saturation (i.e., boiling) temperature at atmospheric pressure (T_b^0) is similar to that of TCA. The values of T_b^0 and other physical properties of isopropanol and TCA are in Table 1. Complete details of the flow reactor design
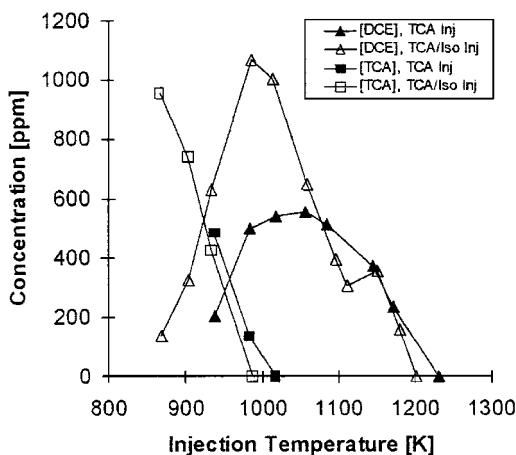
FIG. 2. Centerline residual TCA and DCE concentrations versus injection temperature for dilute sprays of pure TCA and TCA/isopropanol.
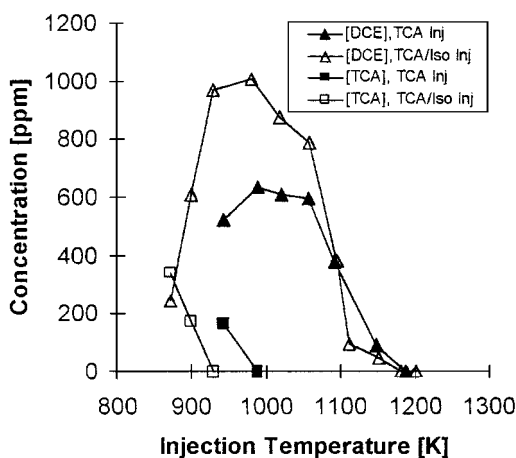

FIG. 3. Centerline residual TCA and DCE concentrations versus injection temperature for dense sprays of TCA and TCA/isopropanol.

and operating characteristics were reported by Higgins [16] and Sgro [17], with more recent modifications detailed by Clack [18].

The spray injector was a prefilming double annular airblast atomizer design used previously [2,3] and described in detail in Ref. [18]. The injector allowed independent control of the mean spray density and droplet size, as measured by interdroplet spacing and Sauter mean diameter, respectively. In the current investigation, only spray density varied between predetermined [3] dense and dilute conditions. The volumetric flow rate of the TCA and TCA/isopropanol sprays was constant at 1 mL/min. The airblast atomizer operated at a gas-to-liquid ratio of 9090 for all sprays. Because the injector hardware experienced elevated temperatures in the postflame region (refer to Fig. 1), it was water cooled to prevent premature vaporization of the injected liquids.

Temperatures at the point of injection varied from 850 to 1200 K and decreased with downstream distance. The equivalence ratio ($\Phi = 0.95$) and residence time (360 ms) were constant for all data. Initial concentrations of TCA after injection and before the start of thermal oxidation were nominally 644 ppm (pure TCA sprays) and 483 ppm (TCA/isopropanol sprays). The nitrogen gas used for airblast atomization produced an additional 0.7% (dense sprays) or 3.2% (dilute sprays) dilution. All species concentrations reflect the total flow of gases in the reactor (air + methane + atomizing N_2). Species concentrations are scaled to the amount of TCA initially injected (scale factor for pure TCA sprays = 1; scale factor for TCA/isopropanol sprays = 1.33).

Results and Discussion

The thermal oxidation of TCA has been studied previously [2–4,21]. At sufficiently high temperatures, TCA undergoes unimolecular decomposition to dichloroethylene (DCE) ($C_2H_2Cl_2$), followed by the bimolecular decomposition of DCE to phosgene ($COCl_2$), hydrogen chloride (HCl), carbon monoxide (CO), and other minor species [4]. The present discussion focuses on TCA, DCE, phosgene, and CO concentrations.

Figures 2 (dilute sprays) and 3 (dense sprays) show the residual TCA and DCE concentrations versus injection temperature at the reactor centerline for both pure TCA and TCA/isopropanol sprays. Both pure TCA and TCA/isopropanol mixtures in dense and dilute sprays show monotonically decreasing residual TCA concentrations with increasing injection temperature. This reflects the absence of TCA formation reactions, leaving TCA concentrations governed only by consumption reactions, whose rates increase with temperature. Residual TCA concentrations for dilute sprays (Fig. 2) also appear to exceed the initial values at injection (644 ppm for pure TCA; 483 ppm for TCA/isopropanol). Such apparently anomalous concentrations are the result of the very long vaporization times that occur at the lowest injection temperatures. Decreasing injection temperature, along with the negative axial temperature gradient of the reactor, increases the time required for complete vaporization of the spray. For a fixed residence time, increasing vaporization times leaves less time to achieve uniform TCA concentrations across the reactor cross section through mixing. The result is a partially mixed vapor core, rich in TCA and located on the reactor centerline. The quartz microprobe, also located on the reactor centerline, extracts samples from this TCA-rich vapor core at the lowest temperatures.

At these low temperatures, differences in vaporization behavior based on spray composition and spray density are also evident. The residual TCA concentrations for TCA/isopropanol sprays are consistently lower than for pure TCA sprays at the same injection temperature (Figs. 2 and 3). The differences in residual TCA concentrations are also more pronounced for dense sprays (Fig. 3) than for dilute sprays (Fig. 2). To a first-order approximation, residual TCA concentrations represent the initial TCA concentration, less the product of the TCA thermal decomposition rate and the fixed residence time within the reactor. As the injection temperature increases, the TCA decomposition rate increases and the residual TCA concentrations decrease. However, this first-order analysis cannot account for the differences in residual TCA concentrations for different spray compositions or different spray densities. Explanation of the observed behavior requires the incorporation of higher-order effects.

The vaporization of the sprays is responsible for the initial gas-phase TCA concentrations and influences the time available for gas-phase thermal decomposition. As discussed previously, at lower injection temperatures, complete vaporization of the spray requires an increasingly larger fraction of the total residence time within the reactor. The longer vaporization times reduce the time available for both mixing and chemical reaction, resulting in a nonuniform radial distribution of TCA concentrations within the reactor. Extractive sampling within such partially mixed flows results in measured residual TCA concentrations that exceed the maximum concentration expected for a fully mixed plug flow (refer to Fig. 2). Therefore, spray vaporization has the potential to influence both time available for chemical reactions and the initial distribution of gas-phase TCA.

The differences in residual TCA concentrations based on spray composition suggest that multicomponent vaporization phenomena are occurring. Droplet vaporization studies often use volatility to parameterize individual constituents within multicomponent fuels. Such studies frequently use the saturation (boiling) temperature at 101.3 kPa (T_b^0) as an indicator of volatility. However the current results, combined with data from our most recent investigation [3], do not support the use of T_b^0 as a measure of the driving force for multicomponent vaporization effects. In a previous study of vaporizing TCA/heptane and TCA/hexadane sprays [3], no multicomponent vaporization effects were evident. The value of T_b^0 for TCA (347 K) is closest to that of isopropanol (356 K, 3% higher) as compared with either heptane (372 K, 7% higher) or hexadecane (560 K, 61% higher) (refer to Table 1). Thus, multicomponent droplet vaporization phenomena should occur, in order of increasing significance, in TCA/isopropanol (smallest ΔT_b^0), TCA/heptane, and TCA/hexadecane (largest ΔT_b^0) sprays for a saturation temperature–based model of volatility. Current and previous [3] data do not support this ordering.

The constant pressure specific heat (C_p) and enthalpy of vaporization (ΔH_v) also are important to the vaporization process. C_p represents the heating rate (K/s) of a droplet for a given heat flux, and ΔH_v represents the amount of energy required to transform a saturated liquid into a saturated vapor at constant pressure. Ranking the four compounds by C_p produces the same ordering as for T_b^0: the value of C_p for isopropanol is closest to that of TCA (9% greater), followed by heptane (56% greater) and hexadecane (247% greater). As with T_b^0, differences in C_p do not appear to correlate with the observed multicomponent vaporization behavior. Ranking the four compounds by ΔH_v produces a different order than those for T_b^0 or C_p. TCA has the smallest value of ΔH_v, followed by heptane (13% greater), isopropanol (40% greater), and hexadecane (148% greater). The values of C_p and ΔH_v for isopropanol are larger than the values for TCA and exceed the TCA values by similar degrees on a percentage basis. Similarly, heptane and hexadecane both have values of C_p and ΔH_v that exceed the TCA values by percentages of the same magnitude. However, the different units of energy for C_p and ΔH_v (J for C_p, kJ for ΔH_v) mean the energy associated with ΔH_v is much larger in absolute value. Assuming TCA/isopropanol sprays initially at room temperature, the energy required to heat TCA or isopropanol to their respective values of T_b^0 is four to six times smaller than the energy required to vaporize either compound at T_b^0. Thus, differences in C_p or T_b^0 constitute much smaller energy differentials than differences in ΔH_v for these hydrocarbon fuels undergoing droplet vaporization. Changes in ΔH_v for multicomponent droplets should therefore have a greater impact on droplet vaporization processes than changes in C_p or T_b^0. For mixtures of compounds with similar T_b^0, the vaporization rate of the mixture should decrease as the mass fraction of the higher ΔH_v constituent increases and should increase as the mass fraction of the lower ΔH_v constituent increases. For mixtures with substantially different values of both T_b^0 and ΔH_v, such as the TCA/hexadecane sprays, the large difference in T_b^0 probably results in separate vaporization events for each component. Solutions from a computational model of vaporizing TCA, TCA/heptane, and TCA/isopropanol droplets (to be published elsewhere) confirm the potential of ΔH_v to promote multicomponent droplet vaporization phenomena. However, the residual TCA concentrations of Figs. 2 and 3 do not reflect the consequences of an increase in ΔH_v accompanying isopropanol addition. The higher ΔH_v of TCA/isopropanol sprays as compared with pure TCA sprays should increase
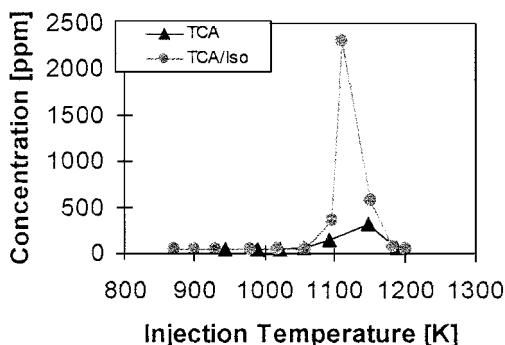
F<small>IG</small>. 4. Representative centerline carbon monoxide (CO) concentrations versus injection temperature for pure TCA and TCA/isopropanol sprays.

vaporization times, thereby reducing mixing and reaction times and increasing residual TCA concentrations. Figures 2 and 3 instead indicate reduced residual TCA concentrations for TCA/isopropanol sprays compared with pure TCA sprays. Evaluation of other potential effects on vaporization, such as changes in equilibrium surface vapor pressure due to changes in droplet surface tension, also did not support the observed results. Without a means of interpreting the observed trends through droplet vaporization processes, the focus turned to droplet flame-holding potential as a function of droplet composition.

The chlorine content of TCA leads to poor combustion characteristics. The substitution of chlorine for hydrogen in TCA reduces the supply of H atoms needed to initiate or propagate combustion reactions. In addition, chlorine scavenges H atoms to form HCl, further reducing the radical population. The addition of normal alkanes to chlorinated hydrocarbon wastes helps mitigate the inhibitory effects of chlorine during combustion. Comparison of the autoignition and flash point temperatures of TCA, isopropanol, heptane, and hexadecane provides a first-order evaluation of the relative combustion characteristics of the four compounds, although much more rigorous approaches are in use [23,24]. The flash point of TCA was not readily found, further proof of its poor combustion characteristics. The flash points for heptane (269 K) and isopropanol (291 K) do not support the residual TCA concentration data. The lower value for heptane indicates that TCA/heptane vapors should ignite more readily, resulting in reduced residual TCA concentrations as compared with pure TCA and TCA/isopropanol sprays under similar conditions. The autoignition temperatures of TCA (810 K), isopropanol (672 K), heptane (496 K), and hexadecane (478 K) would imply similar conclusions, which do not support the current data. However, estimation of the equilibrium vapor pressures of these mixtures does suggest

that ignition of TCA/isopropanol droplets is most likely to occur. Heptane, hexadecane, and isopropanol were all added to TCA in 1:3 mixtures, resulting in a liquid volume fraction for each of 0.25. The liquid mass fractions of each of the unchlorinated additives are also approximately equal: 0.14 for heptane, 0.16 for hexadecane, and 0.16 for isopropanol. However, estimating the equilibrium mole fractions of the unchlorinated additive vapors at the droplet surface by Raoult's law reveals a marked difference: 0.18 for heptane, 0.10 for hexadecane, and 0.30 for isopropanol. The much lower molecular weight of isopropanol leads to a higher liquid- and vapor-phase mole fraction. The higher mole fraction of isopropanol vapor at the droplet surface would increase the likelihood of achieving a minimally combustible gas-phase mixture of TCA, isopropanol, and oxygen at the droplet surface. Based on these data, there appears to be evidence that blending strategies intended to improve combustion and destruction of incineration-resistant wastes should consider ΔH_v for droplet vaporization and molecular weight differences for droplet flame-holding considerations. Experimentally determined equilibrium vapor contours for these mixtures would provide a more accurate representation of the droplet surface composition.

The DCE concentrations presented in Figs. 2 and 3 exhibit differences between pure TCA and TCA/isopropanol sprays as injection temperature increases. TCA/isopropanol sprays produce consistently higher concentrations of DCE than pure TCA sprays at the same temperature. The differences occur for both dense and dilute spray conditions. A marked DCE concentration peak for TCA/isopropanol sprays occurs around 1000 K, whereas the pure TCA sprays reach a lower maximum over a wider range of temperatures. The characteristics of the DCE concentration profiles are largely independent of spray density. These results differ slightly from our previous studies of TCA/alkane thermal oxidation [3], where both pure TCA and TCA/alkane sprays exhibited strong peaks in DCE concentration. In that study [3], only TCA injected as a gas did not exhibit the strong peak in DCE concentration. Peak DCE concentrations of approximately 1100 ppm in both Figs. 2 and 3 agree well with measured peak DCE concentrations in previous TCA/alkane studies [3]. Also, the differences in DCE concentrations between pure TCA and TCA/isopropanol sprays diminish beyond the peak value at higher temperatures. This behavior is also in good agreement with Ref. [3], suggesting that the physical influence of multicomponent droplet vaporization is largely eliminated at higher temperatures.

Carbon monoxide (CO) concentrations (Fig. 4) peak around 1100 K for TCA/isopropanol mixtures. The addition of isopropanol in TCA/isopropanol sprays results in higher peak CO concentrations than
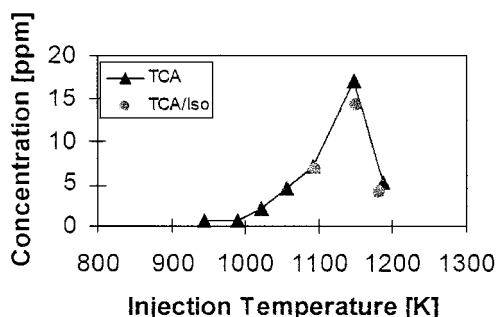
Fig. 5. Representative centerline phosgene ($COCl_2$) concentrations versus injection temperature for pure TCA and TCA/isopropanol sprays.

for pure TCA sprays under the same conditions. Further, the increase in peak CO concentration resulting from incorporating isopropanol is greater than the increase in peak CO concentrations resulting from incorporating heptane in a lean ($\Phi = 0.75$) environment [3]. In a lean ($\Phi = 0.75$) combustion environment, TCA/heptane mixtures produced approximately a twofold increase in peak CO concentrations compared with pure TCA [3]. In a near-stoichiometric ($\Phi = 0.95$) combustion environment, TCA/isopropanol mixtures produced nearly a 10-fold increase in peak CO concentrations compared with pure TCA. The absolute and relative increases in peak CO concentrations likely reflect several phenomena. CO production decreases with increasing equivalence ratio. Therefore, in near-stoichiometric conditions, the percentage increase in peak CO concentrations resulting from isopropanol addition is likely greater because the peak CO concentration for pure TCA is lower. Previous parametric studies of equivalence ratio effects on pure TCA thermal oxidation [2,4] also found decreasing CO concentrations with increasing equivalence ratio. The decomposition of isopropanol itself also likely contributes to the observed CO peak. Previous experiments with heptane and hexadecane [3] found that as much as 50% of the peak CO concentration resulted from oxidation of the alkane. CO generated in the methane/air flame was measured in the background combustion environment at less than 30 ppm and therefore represents a negligible fraction of the peak CO concentrations reported here.

Phosgene ($COCl_2$) concentrations were expected to increase for TCA/isopropanol mixtures relative to pure TCA since isopropanol is an oxygenate, which tends to form oxygenated by-products. However, pure TCA and TCA/isopropanol mixtures exhibit no difference in phosgene concentrations in Fig. 5. The measured phosgene concentrations are in agreement with previous investigations of pure TCA [2] and TCA/alkane [3] mixtures.

Conclusions

Analysis of data taken during the vaporization and thermal decomposition of single-component (TCA) and multicomponent (TCA/isopropanol) polydisperse sprays leads to several findings. Results show increased by-product species concentrations for TCA/isopropanol sprays as compared with pure TCA sprays, in general agreement with previously obtained results for TCA/heptane and TCA/hexadecane. At temperatures below 1000 K, TCA/isopropanol sprays produce lower residual TCA concentrations than pure TCA sprays. This behavior does not reflect preferential vaporization and is not the result of conventional volatility-driven multicomponent droplet vaporization phenomena because the saturation (boiling) temperatures of TCA and isopropanol at 101.3 kPa (T_b^0) are nearly identical. Physical property data, numerical modeling (to be published elsewhere), and previous experimental results for TCA/heptane and TCA/hexadecane all suggest that T_b^0 alone does not adequately characterize multicomponent droplet vaporization under these conditions. Numerical modeling results show that the latent enthalpy of vaporization (ΔH_v) is a more accurate predictor than T_b^0 of these multicomponent, vaporizing sprays. In addition, isopropanol with a lower molecular weight than TCA results in an isopropanol mole fraction in the liquid and vapor phases that is greater than its liquid-phase volume fraction. The higher isopropanol mole fraction in the vapor surrounding the droplet dilutes the TCA and its inhibitory effects on combustion, thereby improving the combustibility of the droplet. These results therefore suggest that molecular weight also be considered when formulating fuel-blending strategies for liquid chlorinated hydrocarbon waste thermal decomposition.

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